RESTEK HPLC

Applicationsnote

HPLC Analysis of Trace-Level Explosives Using Pinnacle II™ C18 and Cyano Columns

Pinnacle II™ high performance liquid chromatography (HPLC) stationary phases were designed to function well under the difficult matrices encountered in environmental samples. The original Pinnacle™ columns served as benchmarks for the selectivity and efficiency of the new Pinnacle II™ columns. While striving to create new columns with characteristics similar to Pinnacle™ columns, Restek also designed the manufacturing process in such a way that they could be priced economically. Using Restek silica, we can go a step further in providing consistent quality and reproducibility.

The new Pinnacle II™ C18 and Cyano columns function as primary and confirmation columns (respectively) to efficiently separate explosives according to US Environmental Protection Agency (EPA) Method 8330A. Environmental methods frequently employ a confirmation column for two reasons. First, many environmental methods require scanning for a large number of related compounds. Because of their similarities, analysts often will encounter coelutions when using a single type of stationary phase. Second, the matrices encountered in many environmental samples can contain components that may interfere or obscure the analytes of interest. By using two columns with different selectivities, analysts can more accurately identify the analytes of interest.

Selectivity for the 14 explosives of interest listed in Method 8330A are similar on the original Pinnacle™ and the new Pinnacle II™ C18 columns (Figure 1). On these C18 columns, there are close-eluting peaks or coelutions for the following compounds: tetryl/nitrobenzene; 2-amino-4,6-dinitrotoluene/4-amino-2,6-dinitrotoluene; and 2,6- dinitrotoluene/2,4dinitrotoluene. Closer examination shows that the new Pinnacle II™ C18 column achieves slightly better resolution for several of these pairs. This may be caused by the slightly higher surface area and carbon load, and the smaller pore volume on the Pinnacle II™ column (110Å) as compared to the Pinnacle™ column (120Å). The higher carbon load of 13% for the Pinnacle II™ column versus 11% for the Pinnacle™ column translates into longer compound retention, better resolution, and column lifetime.

According to Method 8330A, these 14 compounds also need to be analyzed on a Cyano column for confirmation (Figure 2). The change from the reversed phase C18 column to the normal phase Cyano column is fairly easy. The method recommends using the same mobile phase for both columns, which allows a quick changeover from the primary analysis to the confirmation analysis. Because the mobile phase is a simple mixture of water

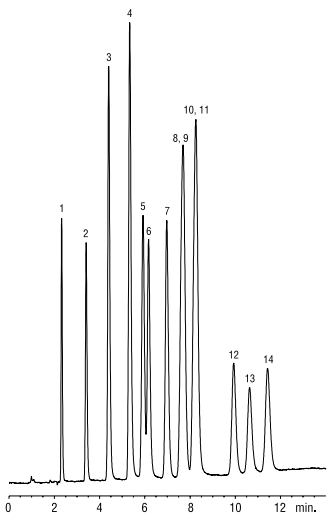
Figure 1

The new Pinnacle II™ C18 column achieves slightly better resolution for difficult to analyze pairs of explosive compounds.

Column: Pinnacle™ C18
Catalog #: 9114575
Dimensions: 250 x 4.6mm
Particle size: 5µm
Pore size: 120Å

Conditions:
Mobile phase: water:methanol (50:50, v/v)
Flow: 1.5 mL/min
Temp.: 27.0°C
Det.: UV @ 254nm

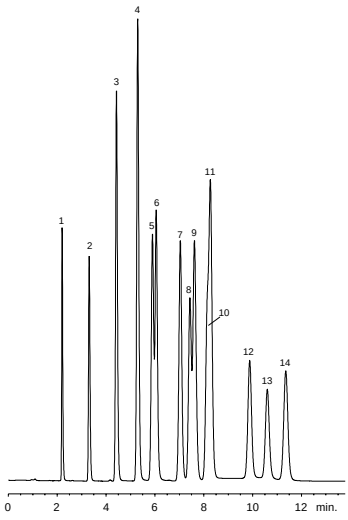
Sample:
Inj.: 3µL
Conc.: 500µg/mL each component
Solvent: acetonitrile
Restek 8330 Calibration mix #1: Cat. # 31450
Restek 8330 Calibration Mix #2 : Cat. # 31451



LC_0194

Peak List:
1. HMX
2. RDX
3. 1,3,5-trinitrobenzene
4. 1,3-dinitrobenzene
5. tetryl
6. nitrobenzene
7. 2,4,6-trinitrotoluene
8. 2-Amino-4,6-dinitrotoluene
9. 4-amino-2,6-dinitrotoluene
10. 2,6-dinitrotoluene
11. 2,4-dinitrotoluene
12. 2-nitrotoluene
13. 4-nitrotoluene
14. 3-nitrotoluene

Column: Pinnacle II C18
Catalog #: 9214575
Dimensions: 250 x 4.6mm
Particle size: 5µm
Pore size: 110Å



LC_0189

HPLC

and methanol, the process of switching from the C18 to the Cyano column is only a matter of removing the primary column and installing the confirmation column on the same HPLC system. There is relatively little downtime, only that required for the column to equilibrate.

Notice that all of the coeluting pairs form the C18 column are resolved from one another on the Cyano column. There is a cluster of compounds: 2-nitrotoluene, 3-nitrotoluene, 4-nitrotoluene, 1,3,5-trinitrotoluene, and 1,3 dinitrotoluene on the Cyano column, but these compounds are well resolved on the C18 column. Again, the selectivity between the original Pinnacle™ Cyano and the new Pinnacle II™ Cyano columns is similar, but the Pinnacle II™ column shows slightly better resolution.

Restek controls the raw material quality from the very beginning of the silica manufacturing process. Add our phase bonding and column packing experience to this high level of quality control, and you benefit from even better column-to-column and analysis-to-analysis reproducibility. Because of this and their economical production, Pinnacle II™ HPLC columns provide a cost-effective analytical tool for many traditional methods used in the environmental industry.

Figure 2

The Pinnacle II™ Cyano column shows slightly better resolution for explosives compounds and is an excellent confirmation column to the Pinnacle II™ C18 for this analysis.

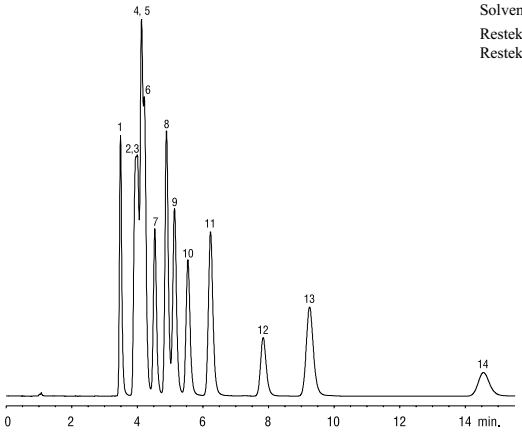
Column: **Pinnacle™ CN**
Catalog #: 9116575
Dimensions: 250 x 4.6mm
Particle size: 5µm
Pore size: 120Å

Conditions:
Mobile phase: water:methanol (50:50, v/v)
Flow: 1.5mL/min
Temp.: 27.0°C
Det.: UV @ 254nm

Sample:
Inj.: 3µL
Conc.: 500 µg/mL each component
Solvent: acetonitrile

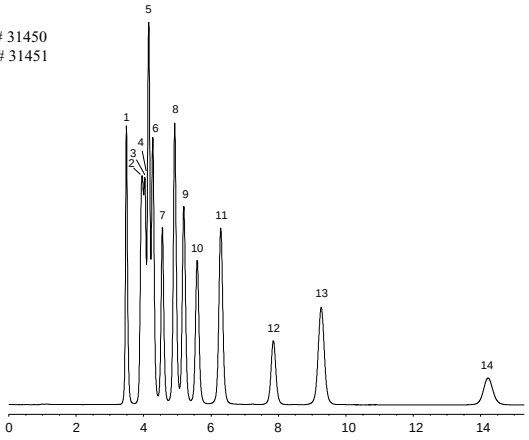
Restek 8330 Calibration mix #1: Catalog # 31450
Restek 8330 Calibration Mix #2 : Catalog# 31451

Peak List:
1. nitrobenzene
2. 2-nitrotoluene
3. 4-nitrotoluene
4. 3-nitrotoluene
5. 1,3-dinitrobenzene
6. 1,3,5-trinitrobenzene
7. 2,6 dinitrotoluene
8. 2,4-dinitrotoluene
9. 2,4,6-trinitrotoluene
10. 4-amino-2,6-dinitrotoluene
11. 2-amino-4,6-dinitrotoluene
12. RDX
13. tetryl
14. HMX



LC_0193

Column: **Pinnacle II CN**
Catalog #: 9216575
Dimensions: 250 x 4.6mm
Particle Size: 5µm
Pore Size: 110Å



LC_0188

Pinnacle II™ C18 5µm Columns

Length	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
50mm	9214551	9214552	9214553	9214555
100mm	9214511	9214512	9214513	9214515
150mm	9214561	9214562	9214563	9214565
250mm	9214571	9214572	9214573	9214575

Pinnacle II™ Cyano 5µm Columns

Length	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
50mm	9216551	9216552	9216553	9216555
100mm	9216511	9216512	9216513	9216515
150mm	9216561	9216562	9216563	9216565
250mm	9216571	9216572	9216573	9216575